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Bromoform Formation in Ozonated
Groundwater Containing
Bromide and Humic Substances

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BROMOFORM FORMATION IN OZONATED GROUNDWATER CONTAINING
BROMIDE AND HUMIC SUBSTANCES

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Abstract

The effect of bromide ion, organic carbon concentration (natural aquatic humic substances), pH, and solar irradiation on the formation of bromoform in ozonated groundwater has been studied. The studies were conducted on four unique samples of groundwater taken from different regions of the Biscayne Aquifer in southern Florida. All other conditions being equal, increases in bromide ion concentrations resulted in increases in CHBr_3 formation. In three of the four samples, CHBr_3 formation decreased as the pH level increased from 5 to 9. The fourth sample exhibited an opposite trend whereby the CHBr_3 concentration increased with increasing pH. Bromoform concentration increased with increased O_3 concentration over an ozone dosage range of 3.4 to 6.7 mg/L.

Ozonated samples placed in sunlight immediately after ozone addition showed a decrease in the formation of CHBr_3 , presumably due to the photodecomposition of $\text{HOBr/OBr}^\cdot$.

Introduction

It has been well established that natural waters, containing aquatic humic substances, form trihalomethanes (THMs) upon chlorination during water treatment. The relative distribution of trihalomethane species, ranging from chloroform to bromoform, is influenced by

several factors, with the dominant factor being the presence (or absence) of bromide ion. Other factors which influence the formation and distribution of THMs include applied chlorine concentration, organic carbon (precursor) concentration, precursor source, chlorine-to-organic carbon ratio, pH, temperature, reaction time, and the presence of chlorine-demanding substances, such as ferrous iron and ammonia (1).

One general strategy for controlling THMs involves the use of alternative oxidants/disinfectants, such as ozone. However, the use of ozone in the presence of bromide and humic species can, under certain conditions, lead to the formation of THMs in the form of bromoform (2). Bromide ion, when present in source water, is not removed by conventional water treatment processes. While the concentration of bromide ion in seawater is 65 mg/L, much lower levels normally are found in freshwaters, with levels ranging from 0 to 2 mg/L (3).

Haag and Hoigné (2,4) studied the ozonation of bromide-containing waters. They found that ozone (O_3) oxidizes bromide ion (Br^-) to hypobromous acid (HOBr) under water treatment conditions. Hypobromite ion (OBr^-) derived from the ionization of HOBr in water further reacts to form either bromate (BrO_3^-) or bromide ion. Eventually, all of the bromide originally present is converted quantitatively to bromate ion. Thus, HOBr/ OBr^- serves as an intermediate in the conversion of bromide to bromate. They found that the maximum transient concentration of the intermediate, HOBr/ OBr^- , occurs under lower pH conditions: the lower the pH, the slower the rate of bromate formation and the greater the maximum concentration of HOBr/ OBr^- as an intermediate. The maximum HOBr/ OBr^- concentration is reached typically within 20 minutes under water treatment conditions (≈ 1 mg/L O_3 , pH 7 to 9). Haag and Hoigné (2,4) also determined that, in the presence of both bromide ion and organic matter such as humic substances, the intermediate HOBr reacts to form organo-bromine compounds. They observed more bromoform formation at lower pH (6.1) than at higher pH (8.8) conditions.

This paper summarizes the results of research conducted to ascertain the effects of organic carbon concentration, bromide ion concentration, pH, and solar irradiation on the formation of $CHBr_3$ in ozonated waters derived from four unique samples taken from different regions of the Biscayne Aquifer. This groundwater system serves as the principal source of drinking water in south Florida, and contains high levels of aquatic humic substances (DOC 5 to 20 mg/L) and moderate levels of bromide ion (150 to 250 μ g/L).

Experimental Methods and Procedures

SAMPLES

Bulk water samples were taken from four different well sites and stored at 4°C until use. The selected sampling sites corresponded to unique regions of the Biscayne Aquifer: samples were taken from

wells situated next to the Alexander Orr water treatment plant, the Hollywood water treatment plant, the John Preston water treatment plant, and the northwest (NW) wellfield. Sodium bromide (NaBr) was added to each water sample in the amount of 1.0×10^{-4} M, corresponding to 8.9 mg/L of bromide ion. The pH of individual aliquots of each sample then was adjusted to either 5.0, 7.0, or 9.0 using H_2SO_4 or NaOH. Screw-cap test tubes (25 x 150 mm, 50 mL capacity) with Teflon-lined caps were used as reactors for both ozonation and bromoform formation.

SAMPLE CHARACTERIZATION

Nonpurgeable organic carbon (NPOC) was measured using a Dohrmann DC-52 Carbon Analyzer. UV absorbance determinations were performed with a Varian 8450A UV-Visible Spectrophotometer. Soluble iron measurements were made using a Perkin-Elmer 403 AA with a graphite furnace and filtered (0.45 μm) samples.

OZONE

Ozone was generated by a standard Tesla coil mounted in an ozone generator tube ("micro-ozonizer", Electrotechnic Products) in an oxygen stream. The generated ozone was carried by Teflon tubing through a silicone rubber stopper to a glass frit diffuser which was submerged in a stoppered test tube for a predetermined time period. Each 50 mL test tube contained 45 mL of sample and 5 mL of headspace. An exit line from the headspace in the reactor carried the gas stream to a secondary trap containing KI solution.

Applied ozone concentrations were determined by filling a test tube reactor itself with KI solution, while utilized ozone concentrations were determined by capturing the vent gas from the reactor in the KI trap and calculating the difference between the amount of applied ozone and the amount of ozone exiting with the vent gas. All ozone doses reported herein correspond to utilized ozone concentration. Total ozone concentrations were determined spectrophotometrically at 352 nm as I_3^- (5).

Typically, ozone was applied to a sample for 40 to 60 seconds, after which the silicone rubber stopper and glass frit were removed and the test tube was sealed with a Teflon-lined cap. All samples were allowed to react with the ozone for four hours, and then the residual ozone/oxidant was reduced by addition of excess sodium thiosulfate.

BROMOFORM ANALYSIS

Concentrations of bromoform (the only trihalomethane species detected) were determined by gas chromatography (Hewlett Packard 5890A/EC Detector) using liquid/liquid extraction with pentane. The extraction procedure involved replacing the Teflon-screw caps with 24 mm Mininert valves and introducing 5 mL of pentane. Control experiments were performed to insure that loss of bromoform to the 5 mL of headspace over the four hour reaction time was insignificant.

IRRADIATION

Light reaction experiments were conducted by placing reaction vials horizontally on the roof of the laboratory for exposure to sunlight in the hours before and after solar noontime. An Eppley Solar-Flux Monitor mounted in the same area was used to measure the light energy associated with these samples. Samples for dark reactions were placed in a test tube rack in a drawer in the laboratory (25°C).

Results and Discussion

Important characteristics of the water samples studied during this research are summarized in Table I. All of the samples contained an ozone demand in the form of ferrous ion, with the lowest overall demand associated with the Orr water treatment plant sample. Moreover, all samples contained ammonia, which can exert a bromine demand. The nonpurgeable organic carbon (NPOC) levels were comparable for three of the samples, with the NW plant sample having a significantly higher level.

TABLE I. CHARACTERISTICS OF GROUNDWATER SAMPLES

Parameter	Sample			
	Orr	Hollywood	Preston	NW
NPOC (mg/L)	6.7	6.7	7.7	15.9
UV Absorbance (254 nm, 1-cm cell)	0.035	0.266	0.353	0.548
Specific Absorbance	0.0052	0.0397	0.0458	0.0345
Ambient pH	7.6	6.7	7.3	8.3
Fe ⁺⁺ (mg/L)	0.05	0.46	0.96	1.45
NH ₃ -N (mg/L)	0.36	1.34	0.79	0.83

It is interesting to note the UV absorbance and specific absorbance determinations: the Orr plant sample contained significantly less UV absorbing material than the other samples, suggesting a much different organic and humic substance composition for this particular sample.

Based on previous work by the authors, ambient bromide levels throughout the Biscayne Aquifer range from approximately 150 to 250 µg/L. Consequently, the amount of bromide ion added externally to the samples (8.9 mg/L) made the ambient levels insignificant. The rationale for spiking water samples with a relatively high level of bromide was

to be able to clearly discern effects and trends in this preliminary research. High bromide levels can occur in freshwater systems by salt water intrusion into over-drafted groundwater basins and drought-impacted freshwater delta systems (6).

The oxidation of bromide ion by ozone, under conditions in which no organic carbon is present, eventually will lead to the quantitative conversion of all bromide ion to bromate ion (BrO_3^-). In the presence of organic carbon (humic substances), some organic-bromine compound formation may occur, depending on pH conditions. Several types of experiments were conducted to define this chemical system for the Biscayne Aquifer, as discussed in the following paragraphs.

The pH dependence of CHBr_3 formation was determined by studying each of the four different samples taken from the Biscayne Aquifer. Samples were ozonated at four ozone concentrations corresponding to utilized O_3 doses of 3.4, 4.7, 5.9, and 6.7 mg/L. Results are presented in Figure 1, portraying the amount of CHBr_3 formed after a 4-hour reaction time period as a function of pH and utilized O_3 dose.

Results derived from three of the groundwater samples generally conformed to the observations of other researchers (4), in which bromoform levels decreased as a function of increasing pH. On the other hand, results derived from one of the samples, the Orr plant sample, exhibited an opposite trend.

One would expect these results to be influenced by competition between reactions leading to the formation of BrO_3^- ion (from O_3 reacting with OBr^- ion) versus reactions leading to the formation of CHBr_3 (from HOBr/OBr^- reacting with organic carbon). At a given pH level, the concentration of CHBr_3 increased as ozone concentration increased for all of the samples. It is interesting to note that, for the Preston plant sample, the lowest utilized ozone dose of 3.4 mg/L resulted in no bromoform formation at any of the pH levels studied. Ostensibly, this behavior may be partially attributable to the presence of an ozone-demanding substance, such as ferrous ion, and a bromine-demanding substance, ammonia-nitrogen.

The most striking behavior observed during these experiments is the apparent anomaly represented by the Orr plant sample, in which high pH conditions, theoretically more favorable for bromate ion formation, lead to higher bromoform levels. This behavior might be attributable to a difference in precursor reactivity associated with this particular source.

In order to elucidate the bromoform formation responses of the different sources, another set of experiments was conducted, in which two samples, those from the Orr and NW plants, were first diluted with organic-free distilled water to several organic carbon concentrations and then spiked with 8.9 mg/L of bromide ion. Each diluted sample was ozonized with 5 mg/L of O_3 (utilized) at a pH of 7.0. The reaction was allowed to proceed for four hours and the resultant CHBr_3 was determined.

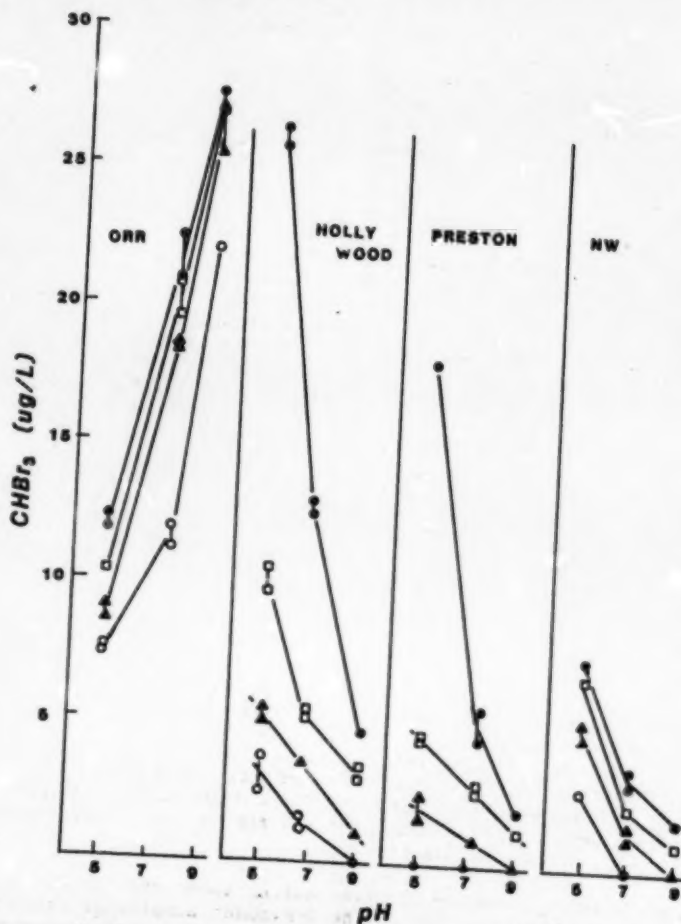


Figure 1. The formation of bromoform in four groundwater samples as a function of pH. Ozone concentration was = 3.4 mg/L, = 4.7 mg/L, = 5.9 mg/L, = 6.7 mg/L.

The results of these experiments are presented in Figure 2. The Orr plant sample, with an initial nonpurgeable organic carbon (NPOC) of 6.7 mg/L, showed a decrease in bromoform formation with decreasing organic carbon level. The NW plant sample, with an initial NPOC of 15.9 mg/L, exhibited a somewhat different response. This sample showed very little change in CHBr_3 formation with the first dilution

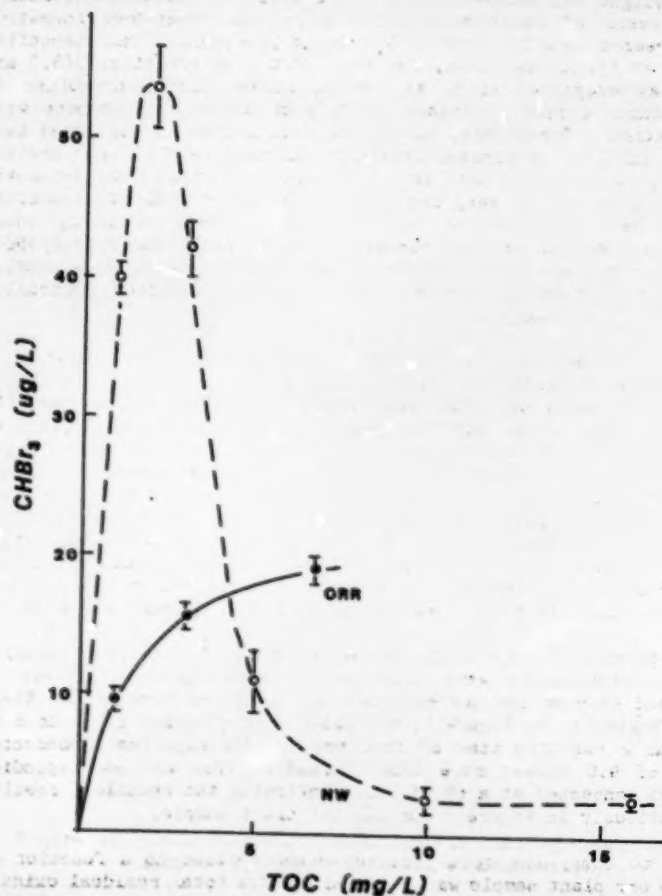


Figure 2. The formation of bromoform in two groundwater samples (NW and ORR) and corresponding dilutions. The formation of bromoform (CHBr₃) is shown in ug/L versus TOC (mg/L). The NW sample shows a sharp peak in bromoform formation at a TOC of approximately 2.5 mg/L, while the ORR sample shows a broader peak at a TOC of approximately 7.5 mg/L. The formation of bromoform decreases as TOC increases beyond these peaks. The data points are shown with error bars. The NW sample is represented by a dashed line and the ORR sample by a solid line.

Haag and Hoigné (4) observed that, for a solution containing constant initial levels of humic acid and bromide ion, bromoform formation first increased as a function of O_3 dose up to a maximum and thereafter decreased at higher O_3 doses, for each of two pH conditions (6.1 and 8.8). They suggested that, at low O_3 doses, little bromoform is formed because direct reactions of O_3 with humic acid compete with HOBr formation. Conversely, at high O_3 doses, they hypothesized that excess O_3 oxidizes brominated intermediates more readily than organic precursors. Since humic acid levels did not vary in their experiments, the trends observed by Haag and Hoigné also would apply to bromoform formation as a function of O_3 /NPOC ratio. Thus, comparing their results with results of this research, the NW plant sample responded in a similar manner, while the Orr plant sample behaved differently, once again suggesting unique precursor characteristics associated with the Orr plant sample.

The effect of bromide ion on bromoform formation was examined in another set of experiments. The results of these experiments appear in Figure 3, based on a utilized ozone dose of 5 mg/L. The NPOC levels for the three samples evaluated were roughly comparable, ranging from 6.7 to 8.3 mg/L. Results associated with two of the samples, from the Hollywood and NW plants, exhibited a response indicative of an oxidant demand. In both of these cases, once this apparent demand was satisfied, $CHBr_3$ formation increased with increasing bromide ion concentration. Conversely, the Orr plant water exhibited no apparent ozone demand and resulted in bromoform formation at all initial bromide ion levels. Of all the samples evaluated, the Orr plant sample contained the lowest levels of Fe^{++} as well as NH_3-N .

In order to examine more fully the behavior of the Orr plant sample, additional experiments were conducted to determine the effects of both pH and bromide ion concentration on bromoform formation. These results, presented in Figure 4, are based on a utilized ozone dose of 5 mg/L and a reaction time of four hours. The experiment conducted at a pH of 9.0 showed more $CHBr_3$ formation than the corresponding experiment conducted at a pH of 7.0, confirming the anomalous results shown previously in Figure 1 for the Orr plant sample.

In order to study the total oxidant concentration as a function of time, the Orr plant sample was ozonated and the total residual oxidant concentrations were determined spectrophotometrically as measured by the oxidation of KI to I_3^- . Bromide ion was added to one sample in the amount of 8.9 mg/L, while no bromide ion was added to the other sample. The results of this experiment are shown in Figure 5.

It can be seen that the addition of bromide ion resulted in significantly higher residual oxidant concentrations over the 30 minute time frame. In the sample without bromide ion, the total residual oxidant concentrations (ozone, peroxides, and related radicals) decreased from 0.8 to almost zero mg/L of Cl_2 equivalents over a 30 min time period. On the other hand, in the corresponding sample with added bromide ion, the total residual oxidant concentrations decreased from 0.8 to 0.45 mg/L Cl_2 equivalents. The behavior of this latter sample is due to the

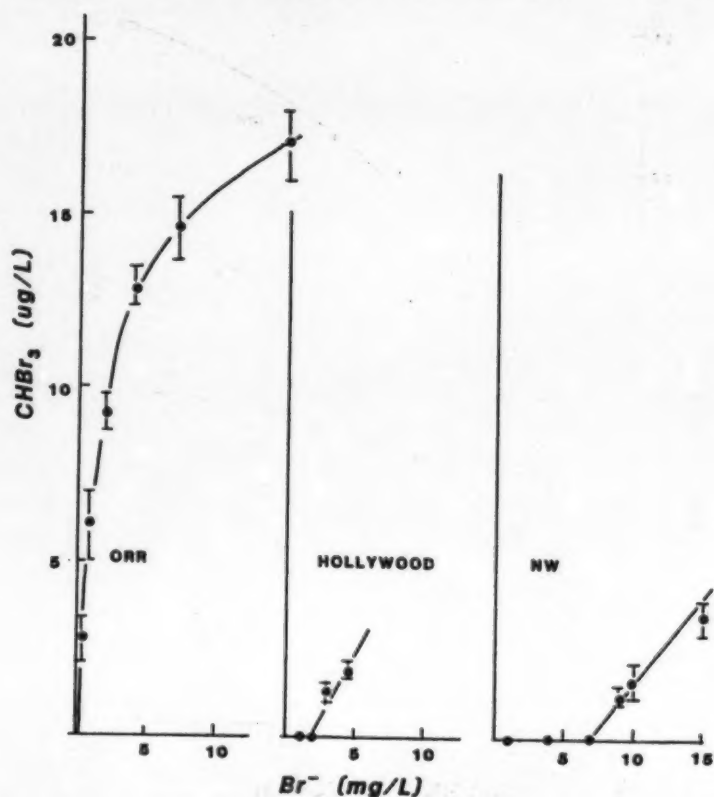


Figure 3. The formation of bromoform in three groundwater samples versus bromide ion concentration.

oxidation of Br⁻ to HOBr/OBr⁻, which is measured as part of the total residual oxidants. It is known that both HOBr and OBr⁻ are sensitive to photodecomposition in sunlight (7). To determine the effect of sunlight on the formation of CHBr₃, two of the water samples were spiked with 8.9 mg/L of bromide ion, ozonated in the laboratory at a utilized ozone dose of 6.0 mg/L, and then each sample was split into two portions. One portion was exposed to sunlight, and the second was kept in the dark. Figure 6 shows the results of the light/dark reactions for the Orr and NW plant samples. It is evident that the formation of CHBr₃ is significantly higher in the light than in the dark for both samples.

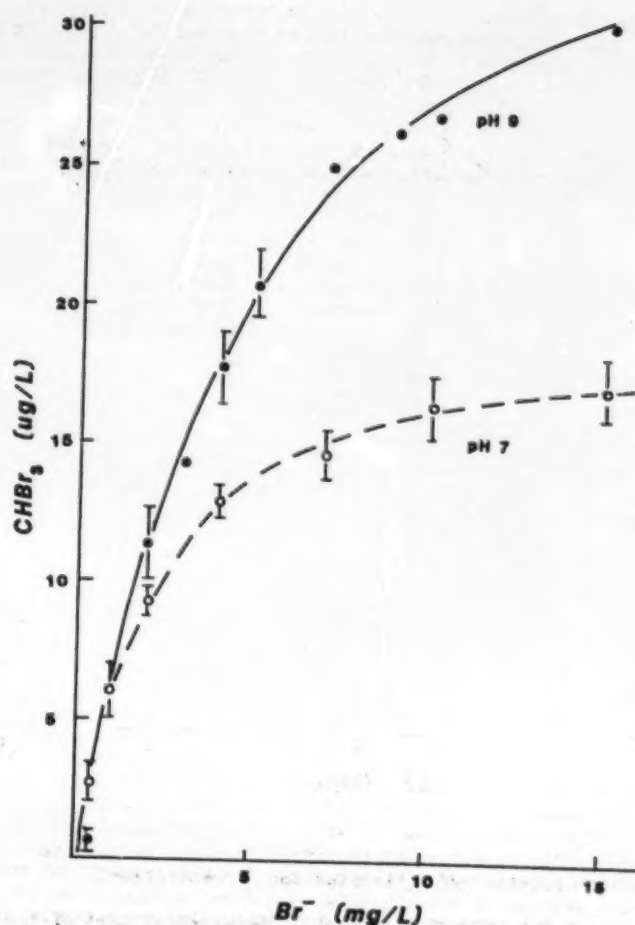


Figure 4. The formation of bromoform versus bromide ion concentration for the Orr plant sample under two pH conditions. For both samples, portions that were ozonated in the lab (6 mg/L utilized O_3) and then exposed to 2.0 watt-hr/m² of light showed a significant decrease in the formation of $CHBr_3$, when compared to the corresponding dark reaction over a four hour reaction time period. In all cases, less bromoform was formed under light reaction conditions as opposed to dark reaction conditions. The NW plant sample, at a pH of 9.0, formed no $CHBr_3$ when exposed to light. These data suggest

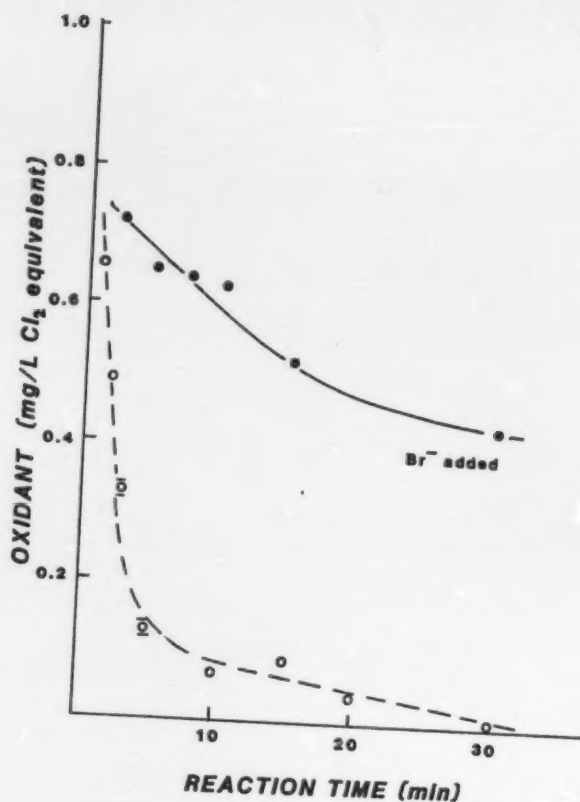


Figure 5. The effect of bromide ion addition on residual oxidant concentration as a function of time for the Orr plant sample.

that for a given water, CHBr_3 can be minimized by immediate exposure of the water to sunlight. Photodecomposition of HOBr/OBr^- and a corresponding reduction in bromoform formation would be expected to occur under water treatment conditions where open contact basins are used.

One complicating factor which deserves additional study is the potential for the incorporation of bromine into organic molecules other than bromoform, both purgeable and nonpurgeable organo-bromine compounds. The photochemical decomposition of HOBr/OBr^- proceeds through free radical reactions (7), and free radical reactions involving halogens are known to produce halogenated organics (7). Thus, more should be

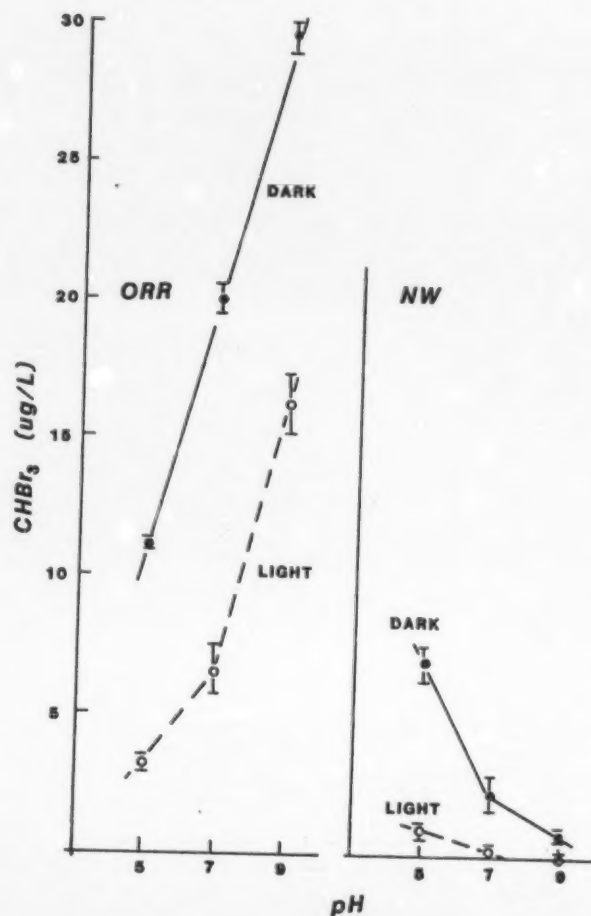


Figure 6. The effect of solar irradiation on bromoform formation as a function of pH for two groundwater samples.

known about the mechanisms and conditions favoring incorporation of bromine into organic molecules before widespread treatment of bromide-containing waters by ozonation and exposure to sunlight.

Conclusions

The results of this research have important implications for water treatment practice. Firstly, the ozonation of waters containing bromide ion and humic substances can lead to the formation of trihalomethanes in the form of bromoform. Although it is unlikely that the current U.S. EPA maximum contaminant level of 100 µg/L will be exceeded, bromoform levels of 10 to 20 µg/L may be formed under some water treatment conditions.

The results of this research and of others (4) indicate that the range of conditions suitable for bromoform formation upon ozonation is considerably narrower than for trihalomethane upon chlorination.

Factors that can be taken into account during design of the water treatment process include the influence of ozone dose on bromoform formation and the photodecomposition of HOBr/OBr⁻ and related effects on bromoform formation.

The reactions of ozone and bromide ion generally are well understood; however the presence of natural aquatic humic substances adds considerable complexity to the system, due to the formation of brominated organic compounds, such as bromoform. Formation of bromoform in ozonated waters is influenced by bromide ion concentration, the source and concentration of humic substances, pH, ozone dose, reaction time, the presence of ozone-demanding substances, and solar irradiation.

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Key Words

Bromoform, Ozonated Groundwater, Bromide Ion, Bromate Ion, Humic Substances, Organic Carbon, Photodecomposition of HOBr/OBr^- , Hypobromous Acid, Hypobromite Ion, Organo-Bromine Compounds.

Résumé

La formation de bromoforme dans les eaux ozonées dépend de la concentration en bromide, de la teneur en carbone lié organiquement (matières humiques naturelles dissoutes dans l'eau), de la valeur du pH, ainsi que de l'exposition à la lumière du jour. Les mesures en question ont été effectuées sur quatre échantillons d'eau souterraine provenant de la région de Biscayne (Floride du Sud). De fortes concentrations en bromide donnèrent lieu à des teneurs élevées en bromoforme. La formation de bromoforme diminua dans trois des quatre échantillons lorsque la valeur du pH passa de 5 à 9. Elle augmenta dans tous les échantillons lorsque le dosage en ozone passa de 3,4 à 6,7 mg/L. Des échantillons ozonés - exposés à la lumière du jour peu après le dosage en ozone - montrèrent une diminution de la formation en bromoforme basée probablement sur la photolyse de HOBr/OBr^- formé.

Zusammenfassung

Die Bildung von Bromoform in ozonierten Grundwassern hängt von der Bromidkonzentration, dem Gehalt an organisch gebundenem Kohlenstoff (natürliche aquatische Huminstoffe), vom pH - Wert sowie von der Belichtung ab. Die entsprechenden Messungen wurden an vier Grundwasserproben aus der Biscayne-Region (südl. Florida) durchgeführt. Erhöhte Bromidkonzentrationen ergaben erhöhte Bromoformbildungen. In drei der vier Proben fiel die Bromoformbildung bei pH Erhöhung von pH 5 auf 9. Sie stieg in allen Proben wenn die Ozondosierung von 3,4 auf 6,7 mg/L erhöht wurde. Ozonte Proben, die kurz nach der Ozondosierung belichtet wurden, zeigten eine Abnahme der Bromoformbildung, die voraussichtlich auf der Photolyse des gebildeten HOBr/OBr^- beruht.

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